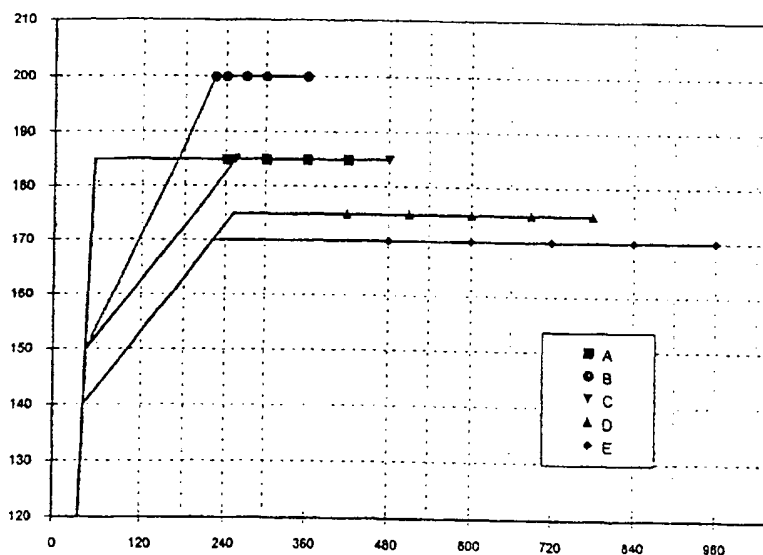




## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/EP99/00940 (22) International Filing Date: 12 February 1999 (12.02.99) (71) Applicant (for all designated States except US): NORSK HYDRO ASA [NO/NO]; Bygdoy Allé 2, N-0240 Oslo (NO). (72) Inventors; and (75) Inventors/Applicants (for US only): TUNDAL, Ulf [NO/NO]; Fagerhaugveien 15, N-6600 Sunndalsøra (NO). ODDVIN, Reiso [NO/NO]; Hoelsandveien 42, N-6600 Sunndalsøra (NO). (74) Agent: BLEUKX, Luc; Hydro S.A., Avenue Marcel Thiry 83, B-1200 Brussels (BE).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  Published With international search report.

(54) Title: ALUMINIUM ALLOY CONTAINING MAGNESIUM AND SILICON



## (57) Abstract

The invention relates to a heat treatable Al-Mg-Si aluminium alloy which after shaping has been submitted to an ageing process, wherein the ageing after cooling of the extruded product is performed in a first stage in which the extrusion is heated with a heating rate above 30°C/hour to a temperature between 100 - 170°C, a second stage in which the extrusion is heated with a heating rate between 5 and 50°C/hour to the final hold temperature between 160 and 220°C and in that the total ageing cycle is performed in a time between 3 and 24 hours.

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**aluminium alloy containing magnesium and silicon**

The invention relates to a heat treatable Al-Mg-Si aluminium alloy which after shaping has been submitted to an ageing process, which includes a first stage in which the extrusion is heated with a heating rate above 30°C/hour to a temperature between 100 - 170°C, a  
5 second stage in which the extrusion is heated with a heating rate between 5 and 50°C/hour to the final hold temperature between 160 and 220°C and in that the total ageing cycle is performed in a time between 3 and 24 hours.

An ageing practise similar to this has been described in WO 95.06759. According to this publication the ageing is performed at a temperature between 150 and 200°C, and the rate  
10 of heating is between 10 - 100°C / hour preferably 10 - 70°C / hour. As an alternative equivalent to this, a two-step heating schedule is proposed, wherein a hold temperature in the range of 80 - 140°C is suggested in order to obtain an overall heating rate within the above specified range.

It is an object of the invention to provide an aluminium alloy which has better mechanical  
15 properties than with traditional ageing procedures and shorter total ageing times than with the ageing practise described in WO 95.06759. With the proposed dual rate ageing procedure the strength is maximised with a minimum total ageing time.

The positive effect on the mechanical strength of the dual rate ageing procedure can be explained by the fact that a prolonged time at low temperature generally enhances the  
20 formation of a higher density of precipitates of Mg-Si. If the entire ageing operation is performed at such temperature, the total ageing time will be beyond practical limits and the throughput in the ageing ovens will be too low. By a slow increase of the temperature to the final ageing temperature, the high number of precipitates nucleated at the low temperature will continue to grow. The result will be a high number of precipitates and mechanical  
25 strength values associated with low temperature ageing but with a considerably shorter total ageing time.

A two-step ageing will also give improvements in the mechanical strength, but with a fast heating from the first hold temperature to the second hold temperature there is substantial chance of reversion of the smallest precipitates, with a lower number of hardening  
30 precipitates and thus a lower mechanical strength as a result. Another benefit of the dual rate ageing procedure as compared to normal ageing and also two step ageing, is that a

slow heating rate will ensure a better temperature distribution in the load. The temperature history of the extrusions in the load will be almost independent of the size of the load, the packing density and the wall thickness' of the extrusions. The result will be more consistent mechanical properties than with other types of ageing procedures.

- 5 As compared to the ageing procedure described in WO 95.06759 where the slow heating rate is started from the room temperature, the dual rate ageing procedure will reduce the total ageing time by applying a fast heating rate from room temperature to temperatures between 100 and 170°C. The resulting strength will be almost equally good when the slow heating is started at an intermediate temperature as if the slow heating is started at room  
10 temperature.

The invention also relates to a Al-Mg-Si-alloy in which after the first ageing step a hold of 1 to 3 hours is applied at a temperature between 130 and 160°C.

- 15 In a preferred embodiment of the invention the final ageing temperature is at least 165°C and more preferably the ageing temperature is at most 205°C. When using these preferred temperatures it has been found that the mechanical strength is maximised while the total ageing time remains within reasonable limits.

- In order to reduce the total ageing time in the dual rate ageing operation it is preferred to perform the first heating stage at the highest possible heating rate available, while as a rule  
20 is dependent upon the equipment available. Therefore, it is preferred to use in the first heating stage a heating rate of at least 100°C / hour.

- In the second heating stage the heating rate must be optimised in view of the total efficiency in time and the ultimate quality of the alloy. For that reason the second heating rate is preferably at least 7°C / hour and at most 30°C / hour. At lower heating rates than 7°C / hour  
25 the total ageing time will be long with a low throughput in the ageing ovens as a result, and at higher heating rates than 30°C / hour the mechanical properties will be lower than ideal.

- Preferably, the first heating stage will end up at 130-160°C and at these temperatures there is a sufficient precipitation of the  $Mg_5Si_6$  phase to obtain a high mechanical strength of the alloy. A lower end temperature of the first stage will generally lead to an increased total  
30 ageing time without giving significant additional strength. Preferably the total ageing time is at most 12 hours.

**Example 1**

Three different alloys with the composition given in Table 1 were cast as Ø95 mm billets with standard casting conditions for AA6060 alloys. The billets were homogenised with a heating rate of approximately 250°C / hour, the holding period was 2 hours and 15 minutes at 575°C, and the cooling rate after homogenisation was approximately 350°C / hour. The logs were finally cut into 200 mm long billets.

**Table 1**

Alloy	Si	Mg	Fe
1	0,37	0,36	0,19
2	0,41	0,47	0,19
3	0,51	0,36	0,19

The extrusion trial was performed in an 800 ton press equipped with a Ø100 mm container, and an induction furnace to heat the billets before extrusion.

In order to get good measurements of the mechanical properties of the profiles, a trial was run with a die which gave a 2 \* 25 mm<sup>2</sup> bar. The billets were preheated to approximately 500°C before extrusion. After extrusion the profiles were cooled in still air giving a cooling time of approximately 2 min down to temperatures below 250°C. After extrusion the profiles were stretched 0.5 %. The storage time at room temperature were controlled to 4 hours before ageing. Mechanical properties were obtained by means of tensile testing.

The mechanical properties of the different alloy aged at different ageing cycles are shown in tables 2-4.

As an explanation to these tables, reference is made to Fig. 1 in which different ageing cycles are shown graphically and identified by a letter. In Fig. 1 there is shown the total ageing time on the x-axis, and the temperature used is along the y-axis.

Furthermore the different columns have the following meaning :

Total time = total time for the ageing cycle.

R<sub>m</sub> = ultimate tensile strength ;

R<sub>P02</sub> = yield strength ;

AB = elongation to fracture ;

Au = uniform elongation .

All these data are the average of two parallel samples of the extruded profile.

**Table 2**

<b>Alloy 1 - 0.36Mg + 0.37Si</b>					
	<u>Total Time [hrs]</u>	<u>Rm</u>	<u>Rp02</u>	<u>AB</u>	<u>Au</u>
A	3	150,1	105,7	13,4	7,5
A	4	164,4	126,1	13,6	6,6
A	5	174,5	139,2	12,9	6,1
A	6	183,1	154,4	12,4	4,9
A	7	185,4	157,8	12,0	5,4
B	3,5	175,0	135,0	12,3	6,3
B	4	181,7	146,6	12,1	6,0
B	4,5	190,7	158,9	11,7	5,5
B	5	195,5	169,9	12,5	5,2
B	6	202,0	175,7	12,3	5,4
C	4	161,3	114,1	14,0	7,2
C	5	185,7	145,9	12,1	6,1
C	6	197,4	167,6	11,6	5,9
C	7	203,9	176,0	12,6	6,0
C	8	205,3	178,9	12,0	5,5
D	7	195,1	151,2	12,6	6,6
D	8,5	208,9	180,4	12,5	5,9
D	10	210,4	181,1	12,8	6,3
D	11,5	215,2	187,4	13,7	6,1
D	13	219,4	189,3	12,4	5,8
E	8	195,6	158,0	12,9	6,7
E	10	205,9	176,2	13,1	6,0
E	12	214,8	185,3	12,1	5,8
E	14	216,9	192,5	12,3	5,4
E	16	221,5	196,9	12,1	5,4

**Table 3**

<b>Alloy 2 - 0.47Mg + 0.41Si</b>					
	<u>Total Time [hrs]</u>	<u>Rm</u>	<u>Rp02</u>	<u>AB</u>	<u>Au</u>
A	3	189,1	144,5	13,7	7,5
A	4	205,6	170,5	13,2	6,6
A	5	212,0	182,4	13,0	5,8
A	6	216,0	187,0	12,3	5,6
A	7	216,4	188,8	11,9	5,5
B	3,5	208,2	172,3	12,8	6,7
B	4	213,0	175,5	12,1	6,3
B	4,5	219,6	190,5	12,0	6,0
B	5	225,5	199,4	11,9	5,6
B	6	225,8	202,2	11,9	5,8
C	4	195,3	148,7	14,1	8,1
C	5	214,1	178,6	13,8	6,8
C	6	227,3	198,7	13,2	6,3
C	7	229,4	203,7	12,3	6,6
C	8	228,2	200,7	12,1	6,1
D	7	222,9	185,0	12,6	7,8
D	8,5	230,7	194,0	13,0	6,8
D	10	236,6	205,7	13,0	6,6
D	11,5	236,7	208,0	12,4	6,6
D	13	239,6	207,1	11,5	5,7
E	8	229,4	196,8	12,7	6,4
E	10	233,5	199,5	13,0	7,1
E	12	237,0	206,9	12,3	6,7
E	14	236,0	206,5	12,0	6,2
E	16	240,3	214,4	12,4	6,8

Table 4

Alloy 3 - 0.36Mg + 0.51Si					
	<u>Total Time [hrs]</u>	<u>Rm</u>	<u>Rp02</u>	<u>AB</u>	<u>Au</u>
A	3	200,1	161,8	13,0	7,0
A	4	212,5	178,5	12,6	6,2
A	5	221,9	195,6	12,6	5,7
A	6	222,5	195,7	12,0	6,0
A	7	224,6	196,0	12,4	5,9
B	3,5	222,2	186,9	12,6	6,6
B	4	224,5	188,8	12,1	6,1
B	4,5	230,9	203,4	12,2	6,6
B	5	231,1	211,7	11,9	6,6
B	6	232,3	208,8	11,4	5,6
C	4	215,3	168,5	14,5	8,3
C	5	228,9	194,9	13,6	7,5
C	6	234,1	206,4	12,6	7,1
C	7	239,4	213,3	11,9	6,4
C	8	239,1	212,5	11,9	5,9
D	7	236,7	195,9	13,1	7,9
D	8,5	244,4	209,6	12,2	7,0
D	10	247,1	220,4	11,8	6,7
D	11,5	246,8	217,8	12,1	7,2
D	13	249,4	223,7	11,4	6,6
E	8	243,0	207,7	12,8	7,6
E	10	244,8	215,3	12,4	7,4
E	12	247,6	219,6	12,0	6,9
E	14	249,3	222,5	12,5	7,1
E	16	250,1	220,8	11,5	7,0



Based upon these results the following comments apply.

5 The ultimate tensile strength (UTS) of alloy no. 1 is slightly above 180 MPa after the A - cycle and 6 hours total time. The UTS values are 195 MPa after a 5 hours B - cycle, and 204 MPa after a 7 hours C - cycle. With the D - cycle the UTS values reaches approximately 210 MPa after 10 hours and 219 MPa after 13 hours.

With the A - cycle alloy no. 2 show a UTS value of approximately 216 MPa after 6 hours total time. With the B - cycle and 5 hours total time the UTS value is 225 MPa. With the D - cycle and 10 hours total time the UTS value has increased to 236 MPa.

10 Alloy no. 3 has an UTS value of 222 MPa after the A-cycle and 6 hours total time. With the B - cycle of 5 hours total time the UTS value is 231 MPa. With the C - cycle of 7 hours total time the UTS value is 240 MPa. With the D - cycle of 9 hours the UTS value is 245 MPa. With the E - cycle UTS values up to 250 MPa can be obtained

15 The total elongation values seem to be almost independent of the ageing cycle. At peak strength the total elongation values, AB, are around 12%, even though the strength values are higher for the dual rate ageing cycles.

**Claims**

1. A heat treatable Al-Mg-Si aluminium alloy which after shaping has been submitted to an ageing process, characterized in that the ageing after cooling of the extruded product is performed in a first stage in which the extrusion is heated with a heating rate above 30°C/hour to a temperature between 100 - 170°C, a second stage in which the extrusion is heated with a heating rate between 5 and 50°C/hour to the final hold temperature between 160 and 220°C and in that the total ageing cycle is performed in a time between 3 and 24 hours.
2. Aluminium alloy according to any one of the preceeding claims, modified in that after the first ageing step a hold of 1 to 3 hours is applied at a temperature between 130 and 160°C.
3. Aluminium alloy according to any one of the preceeding claims, characterized in that the final ageing temperature is at least 165°C.
4. Aluminium alloy according to any one of the preceeding claims, characterized in that the final ageing temperature is at most 205°C.
5. Aluminium alloy according to any one of the preceeding claims, characterized in that in the first heating stage the heating rate is at least 100°C / hour.
6. Aluminium alloy according to any one of the preceeding claims, characterized in that in the second heating stage the heating rate is at least 7°C / hour.
7. Aluminium alloy according to any one of the preceeding claims, characterized in that in the second heating stage the heating rate is at most 30°C / hour.
8. Aluminium alloy according to any one of the preceeding claims, characterized in that at the end of the first heating step the temperature is between 130 and 160°C.

9. Aluminium alloy according to any one of the preceeding claims,  
c h a r a c t e r i z e d   i n   t h a t   the total ageing time is at least 5 hours.
  
10. Aluminium alloy according to any one of the preceeding claims,  
c h a r a c t e r i z e d   i n   t h a t   the total ageing time is at most 12 hours.

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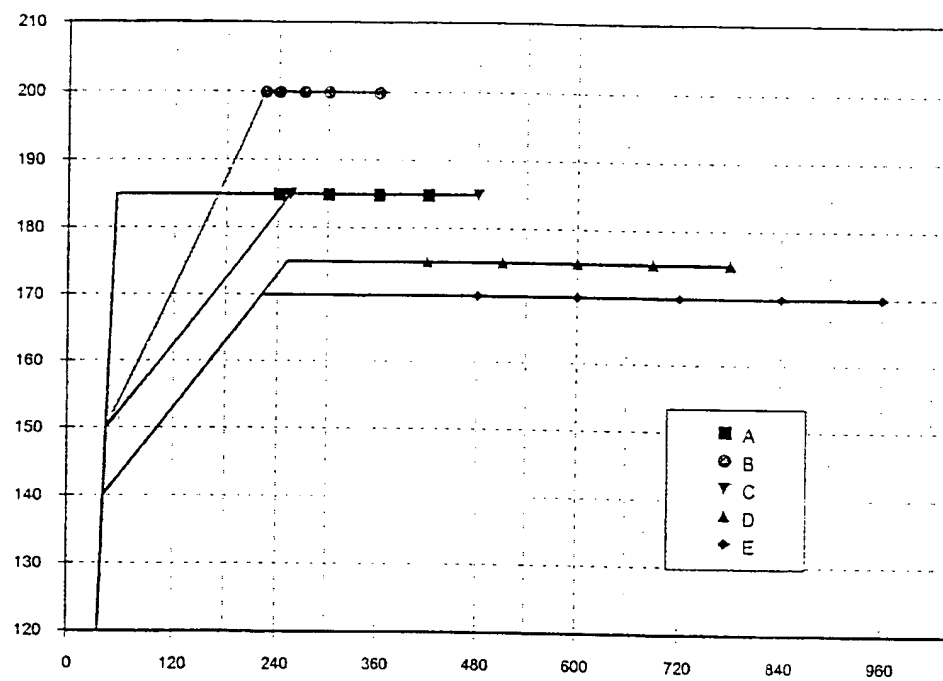


FIG. 1

## INTERNATIONAL SEARCH REPORT

International Application No.

PCT/EP 99/00940

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 7 C22F1/05

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C22F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 95 06759 A (ALCAN INT LTD ;YIU HANG LAM (GB); RICKS RICKY ARTHUR (GB); COURT S) 9 March 1995 (1995-03-09) cited in the application , sentence 15 - sentence 31; figure 6 ---	1-10
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☐ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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